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Silicon Backbond Strain Effects on NH_3 Surface Chemistry - Si(111)-(7x7) Compared to Si(100)-(2x1)

by

P.J. Chen, M.L. Colaianni and J.T. Yates, Jr.

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Surface Science Center
Department of Chemistry
University of Pittsburgh
Pittsburgh, PA 15260

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P. J. Chen#, M. L. Colaianni and J. T. Yates, Jr.

Surface Science Center
Department of Chemistry
University of Pittsburgh
Pittsburgh, PA 15260

#Department of Physics, University of Pittsburgh, Pittsburgh, PA 15260

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Abstract

The role of surface structure in controlling NH_3 surface chemistry has been investigated by HREELS and TPD on Si(111)-(7x7) and Si(100)-(2x1). Following the initial NH_3 dissociative adsorption producing NH_2(a) and H(a), the production of >NH(a) species is favored on Si(111)-(7x7) between 300-600 K. This is believed to occur through the insertion of >NH(a) into the backbond of the Si adatom on Si(111)-(7x7) and to be driven by the relief of bond strain on the Si adatom backbonds. By contrast, this stepwise reaction mechanism is absent on Si(100)-(2x1). Instead, the surface NH_2(a) species remain thermally stable until ~600 K, when a new reaction channel opens up leading to the partial depletion of NH_2(a) by recombination with H(a) to form NH_3(g).

I. Introduction

The reaction of NH_3 with Si surfaces has been extensively studied by various surface science techniques. It has been fairly well understood that the existence of Si dangling bonds on the surface is responsible for the initial low temperature reactivity of the surface [1-5]. The initial stage of NH_3 reaction with a Si surface proceeds until the saturation of the Si surface dangling bonds is achieved, which gives rise to the apparent saturation behavior observed experimentally. Both ultra-violet photoelectron spectroscopy (UPS) and atom-resolved scanning tunneling microscopy (STM) studies have shown the suppression of the dangling bond electronic states upon NH_3 adsorption. Surface vibrational spectroscopic measurements [6-10] performed on Si(100)-(2x1), Si(111)-(7x7) and amorphous Si surfaces indicate that mainly Si-H and $Si-NH_2$ species are initially produced through the dissociative adsorption of NH_3. Thus the low temperature surface chemistry primarily involves the formation of Si-N< and Si-H chemical bonds localized on the dangling bond sites. However, of equal interest and importance is how such a surface reaction <u>proceeds</u> by continued bond breaking and bond making on the surface as the surface temperature is raised. This involves understanding the evolution of reaction intermediates on the surface and most important of all, the role surface structure plays in dictating these processes. Despite the underlying fundamental and technological importance, these aspects of Si surface chemistry have gained little microscopic level understanding due largely to the degree of experimental difficulty and the demanding task to perform high-

level quantum chemical calculations. Issues concerning the intermediate stages of surface reaction on silicon remain largely unsettled even for a relatively simple adsorbate such as NH_3.

In this paper, we investigate the role of surface structure in controlling Si surface chemistry by conducting a comparative study of NH_3 chemistry on Si(111)-(7x7) and Si(100)-(2x1) surfaces under identical experimental conditions. By performing species-specific vibrational spectroscopic measurements as a function of temperature, we have been able to follow the evolution of surface reaction intermediate species produced by NH_3 adsorption.

II. Experimental

The experiments were conducted in a UHV system equipped with a high resolution electron energy loss spectrometer (HREELS) and a shielded quadrupole mass spectrometer (QMS) with apertured line-of-sight detection capability for temperature programmed desorption (TPD) studies. Both the Si(111) and Si(100) crystals were p-type boron-doped (10 Ωcm resistivity) wafers oriented within 0.5° to their respective crystallographic directions and prepared following standard procedures described in detail elsewhere [10]. Exposure to NH_3 gas was conducted through a calibrated effusive gas doser which contains an internal micron-sized pinhole and a front-mounted microcapillary-array for collimation purpose. The crystal temperature was controlled with a microprocessor-based power supply which provided a reproducible linear temperature ramp for TPD measurements.

III. Results and Discussion

The comparison of HREEL spectra taken from NH_3-exposed Si(111) and Si(100) surfaces is shown in Figure 1. Both sets of spectra were taken following the same initial NH_3 exposure of $4x10^{14}$ molecules/cm^2 at 100 K and then sequentially heating to the specified temperatures. The exposure level and temperature are chosen to ensure the saturation of surface Si dangling bonds on both surfaces and to eliminate any weakly bound molecular NH_3. On the Si(100) surface, only two types of surface species are identified, i.e., -H and $-NH_2$. The spectrum and the surface species are virtually unchanged on Si(100) by annealing from 300 K to 600 K with the exception of a slight intensity reduction in the NH_2 stretching mode. On the Si(111) surface, the 300 K spectrum is very similar to that obtained on Si(100) with the exception of a small shoulder at ~1100 cm^{-1}. Once again, at 300 K the (majority) surface species are -H and $-NH_2$, in agreement with previous studies. By annealing the Si(111) surface to 600 K, extensive changes take place in the vibrational spectrum in marked contrast to the behavior on Si(100). The $-NH_2$ deformation mode at 1550 cm^{-1} is strongly attenuated, while a new mode becomes fully developed at 1100 cm^{-1}. We have identified this 1100 cm^{-1} mode as the deformation mode, δ(NH), of a surface >NH species in a detailed vibrational study published elsewhere [10]. This >NH species is directly derived from further dissociation of $-NH_2$ on the surface. This <u>stepwise</u> dissociation of $-NH_2$ into >NH and H is also indicated

by a marked intensity increase for the Si-H stretching mode at 2100 cm^{-1} for the Si(111) surface.

The comparison of desorption products from the Si(100) and Si(111) surfaces provides additional information about the surface reaction derived from NH_3 dissociation. For this purpose, a set of comparative line-of-sight TPD measurements was made on the two Si surfaces. The results are shown in Figure 2. Below 200 K, the desorption of weakly bound molecular NH_3 is the common feature from both surfaces. When the surface temperature is raised to ~600 K, an associative desorption reaction producing NH_3 is observed on Si(100) (hatched region). This is consistent with the vibrational observation that the $-NH_2$ species persist on this surface at this temperature. When the surface temperature is sufficiently high, the recombination of NH_2(a) with H(a) occurs on the surface, and the desorption of NH_3 occurs. Taylor et al. [5] demonstrated in an isotope experiment that surface NH_2 is indeed responsible for the observed recombination channel which primarily desorbs NH_2D and NH_3 species near 600 K on a partially D-covered Si(100) surface. By contrast, as shown Figure 2B, this associative NH_3 desorption channel does not exist on the Si(111)-(7x7) surface. Vibrational observation of the production of surface >NH species in the temperature range 300 - 600 K indicates that, unlike Si(100), there exists an efficient pathway for the further dissociation of surface $-NH_2$ into >NH and -H on Si(111). The direct kinetic effect of this is the suppression of the associative desorption channel to form NH_3(g) from NH_2(a) and H(a).

We have established thus far that following the initial low

temperature dissociation of NH_3 into NH_2(a) and H(a), there are two distinct pathways for the surface reaction to further proceed. On Si(111), an efficient dissociation channel exists for NH_2(a) to produce stable surface >NH and -H species between 300 and 600 K. On the Si(100) surface, this dissociation channel for chemisorbed NH_2 is absent. Instead, a remarkable thermal stability is found for the NH_2(a) species on this surface, which leads to the opening up of the associative NH_3 desorption channel starting at ~600 K. Such a contrasting behavior suggests strongly that the surface structure of the two silicon crystals plays an important role. To understand the underlying mechanism that accounts for the observed differences, a detailed look into the surface atomic and electronic structure on both surfaces is presented next.

First we turn to the Si(111)-(7x7) surface. The spectroscopic observation of a stable >NH species at moderate temperatures suggests that the activation barrier for the chemisorbed NH_2 dissociation is sufficiently low so that >NH becomes the predominant species present on the surface at 600 K (Figure 1). Note that even at 300 K the vibrational spectrum indicates the presence of a small amount of >NH on the surface. Furthermore, the ability for the surface to accommodate NH(a) + 2H(a) species without any desorption taking place in this temperature range indicates that substrate Si-Si bond breaking is not only involved but is also thermodynamically favorable.

Wolkow and Avouris [4,5] have presented atom-resolved STM studies of the NH_3 reaction on Si(111)-(7x7) at room temperature. Reaction was found to occur on the Si rest atom and adatom dangling

bond sites, and a higher reactivity was observed for the rest atoms. Therefore, upon the saturation of surface dangling bonds by NH_3 adsorption, NH_2(a) and H(a) are produced and localized at the adatom and rest atom sites. The localization of these adsorbate fragments and the open structure of the Si(111)-(7x7) minimizes steric interaction, even for monolayer coverage. Overall, the preservation of the (7x7) reconstruction as seen from STM indicates no extensive Si-Si bond breaking, consistent with our spectroscopic observation that the majority surface species are NH_2(a) and H(a). In the following discussion, we will ignore the contribution from the Si atom associated with the corner hole on the Si(111)-(7x7) unit cell and concentrate only on the adatoms and rest atoms which account for over 90 percent of the surface dangling bonds. Note that while the formation of a $-NH_2$ species involves bonding to only a single surface Si atom, a fully coordinated >NH species necessarily involves bonding to two Si atoms, i.e., the insertion of a N atom between two Si atoms. Given the fact that a NH_2(a) species always forms on a Si dangling bond site, and comparing the length of a typical Si-N bond (~2.0 Å) to the large distance separating surface Si dangling bonds (~5 Å), it is clear that a >NH can not bridge between two Si dangling bond sites [11]. This limits the >NH formation only to the nearest neighbor Si atoms, i.e., >NH formation requires the breaking of a Si-Si backbond.

With this structural model in mind, we next examine the nature of the Si-Si backbonds on the Si(111)-(7x7) surface. Since the surface Si atoms can be classified into adatoms and rest atoms, the Si-Si backbonds associated with them can also be classified into

adatom backbonds and rest atom backbonds (Figure 3a). While both types of backbonds involve triply coordinated surface Si atoms, there is in fact a significant difference between these two types of silicon backbonds in that the adatom backbonds are strained while the rest atom backbonds are not [12,13]. The important role of these strained (and therefore weaker) Si backbonds in controlling surface chemistry has been recognized by Boland [14]. In STM investigations of hydrogen interaction with the Si(111)-(7x7) surface, Boland [14] presents compelling experimental evidence that the strained adatom backbond exhibits a significantly lower reaction barrier toward atomic hydrogen than the unstrained rest atom backbond. The relaxation of the strain on these adatom backbonds is responsible for a reduced barrier to reactive Si-Si bond breaking favoring the formation of SiH_x (x>1) species on Si adatoms. This property pertains to the Si adatoms that form the (7x7) reconstruction. Based on the above analysis, an analogous adatom backbond strain relaxation mechanism can be proposed for the formation of a >NH species, as schematically depicted with a ball-stick model in Figure 3c. The opening of the original Si four-member ring through the insertion of N into the Si backbond (Figure 3c) should facilitate the relief of strain on all three backbonds. We propose that this is most likely the structural origin of the stable surface >NH species specifically produced from NH_3 between 300 and 600 K on Si(111)-(7x7). Quantitatively, since the Si adatoms outnumber the Si rest atoms 2 to 1 in the (7x7) reconstruction, a similar fraction of surface $-NH_2$ species should be associated with the Si adatoms upon initial saturation of the Si

dangling bonds by dissociative NH_3 adsorption. The presence of a minority $-NH_2$ species detected by HREELS (Figure 1B) at 600 K may be attributed to the surface NH_2(a) associated with the Si rest atoms.

Figure 4 shows the simpler Si(100)-(2x1) surface. The clean surface (2x1) reconstruction consists of σ-bonded Si dimer rows formed between adjacent surface Si atoms and two dangling bond orbitals at each end of the Si dimer which overlap (in π-bond fashion) weakly with each other (Figure 4a). Spectroscopic observation of chemisorbed NH_2 and H following initial dissociative NH_3 adsorption indicates that, similar to the situation on Si(111)-(7x7), only the surface dangling bonds are involved while the Si dimer σ-bond remains intact (Figure 4b). This is consistent with LEED observation that the (2x1) periodicity is preserved after initial surface saturation and also with the detection of this NH_2 structure by ESDIAD [15]. The formation of a fully-coordinated >NH species on the surface would require the insertion of the >NH unit between two Si atoms. Here the structural constraint may come from the large separation between dimer units so that any possible >NH formation is limited to within a single dimer unit. Although the Si dimer bond is expected to be the weakest of all the Si-Si bonds and it is not completely free of lattice strain [16], unlike the Si-Si backbond on Si(111)-(7x7), it apparently survives further surface reaction and remains intact up to 600 K, as shown by the absence of >NH formation. Currently, there is no rigorous theoretical structural study available for direct comparison. Nevertheless, our experimental results do not support the claim of

a recent cluster model [17] which predicts a very low (~0.8 eV) activation barrier for the further dissociation of NH_2(a) into NH(a) and H(a) on Si(100)-(2x1) surface.

After the exclusion of a stable >NH species on the Si(100) surface, it is rather straightforward to understand the origin of the associative NH_3 desorption process which is specifically observed on this surface. The stability of NH_2(a) on Si(100)-(2x1) permits recombination with H(a) to be energetically favored compared to >NH + -H production. Note that a <u>transient</u> >NH* species might still be involved in the NH_2 dissociation process above 600 K. But a stable surface >NH species as a reaction intermediate is never observed spectroscopically under any temperature or coverage conditions.

We conclude by noting that, by virtue of the localized adsorbate-substrate chemical bond and a high barrier to surface diffusion, the dissociation pathway for NH_2(a) is greatly influenced by surface structure. Compared to a metal surface, the fundamental difference for silicon is the active participation of a substrate chemical bond in the dissociation process. This is reflected in the fact that, instead of relying on the existence of vacant adsorption sites, the NH_2(a) dissociation process on Si(111)-(7x7) itself involves breaking of the substrate Si-Si bond to create new adsorption sites locally to accommodate the dissociation products. Therefore, it is the overall energy balance of <u>all</u> the chemical bonds involved that determines the feasibility of a particular dissociation pathway. This ultimately relates to surface structure and bonding issues such as the bond strength

between the surface and bulk Si atoms, the lattice strain associated with each individual bond, the propagation of the strain field to the neighboring silicon atoms and so forth.

IV. Conclusion

In summary, we have demonstrated that NH_3 chemistry on Si surfaces is dominated by the detailed surface atomic and electronic structure. Precise knowledge about the surface reconstruction and bonding leads to the understanding of the elementary steps chosen for a surface chemical process. The low temperature surface chemistry of NH_3 on silicon is largely related to the presence of surface dangling bonds. In the case of NH_3 adsorption at low temperatures on Si(111)-(7x7) and Si(100)-(2x1), this only results in a limited degree of reaction, leading to the formation of $-NH_2$ and -H surface species. At elevated temperatures, the breaking of substrate Si-Si bonds becomes the key process to advance the surface reaction. The selective breaking of substrate Si-Si bond at this stage of surface reaction depends crucially on the surface structure and detailed local bonding of the Si atoms. Certain strained Si-Si bonds, such as the adatom backbonds on Si(111)-(7x7), are weaker than the bulk Si-Si bonds and are therefore more subject to reactive bond breaking. In particular, the relief of the strain on the remaining Si adatom backbonds may be achieved by the insertion of an >NH species into a Si-Si backbond. This is believed to specifically provide the driving force for the further dissociation of NH_2(a) on the Si(111)-(7x7) adatom sites between 300 and 600 K. By contrast, this stepwise reaction mechanism is

absent on the Si(100)-(2x1) surface because no such favorable surface conditions exist. Instead, the $NH_2(a)$ species remain thermally stable until ~600 K, when a new reaction channel opens up leading to the partial depletion of $NH_2(a)$ by recombination to form $NH_3(g)$. These concepts likely apply to the behavior of many adsorbates on the various silicon single crystal surfaces.

Acknowledgement

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Figure Captions

Figure 1. HREEL spectra from (a) NH_3/Si(100)-(2x1) after sequential heating to 300 and 600 K. The spectra were recorded at a primary energy of 4.2 eV; (b) NH_3/Si(111)-(7x7) after sequential heating to 300 and 600 K. The spectra were recorded at a primary energy of 6.1 eV. The NH_3 exposure on both surfaces were $4x10^{14}$ cm^{-2} at 100 K.

Figure 2. Temperature programmed desorption mass spectra of H_2 (m/e=2) and NH_3 (m/e=17) from (a) Si(100)-(2x1) and (b) Si(111)-(7x7) after an identical NH_3 exposure of $4x10^{14}$ cm^{-2}. The heating rates were 1 K/sec on both surfaces.

Figure 3. (a) Ball-stick model for the Si adatom and Si rest atom on Si(111)-(7x7). Note the difference in the two types of Si-Si backbonds associated with the two types of Si surface sites. (b) Adsorption geometry for $-NH_2$ and -H. (c) Proposed structure for the stable surface >NH species.

Figure 4. (a) Top view of the Si(100)-(2x1) surface. (b) Ball-stick model of the adsorption configuration of $-NH_2$ and -H on Si(100)-(2x1).

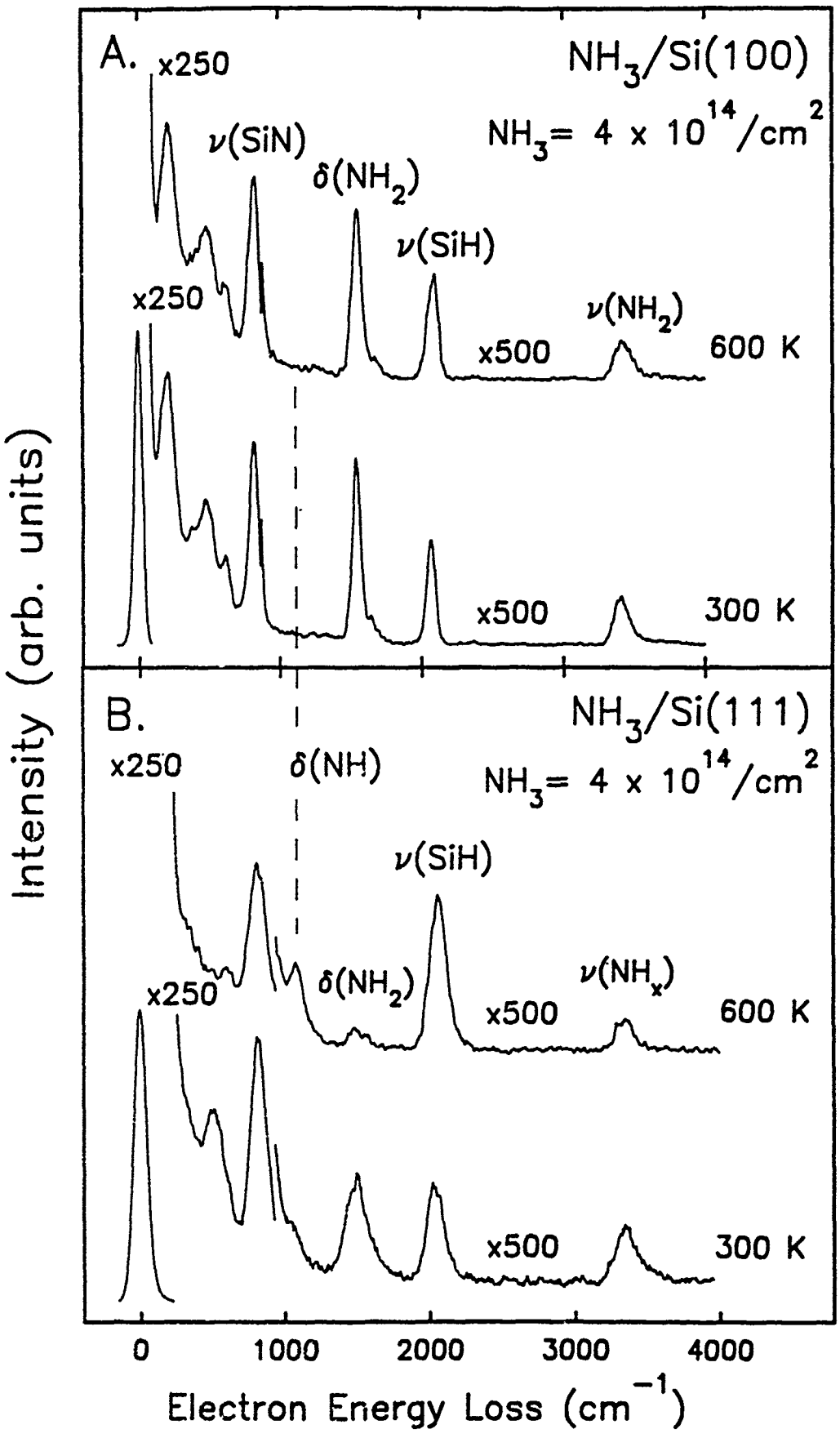

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Figure 1

Comparison of Thermal Desorption Products from NH_3 on Si(100) and Si(111)

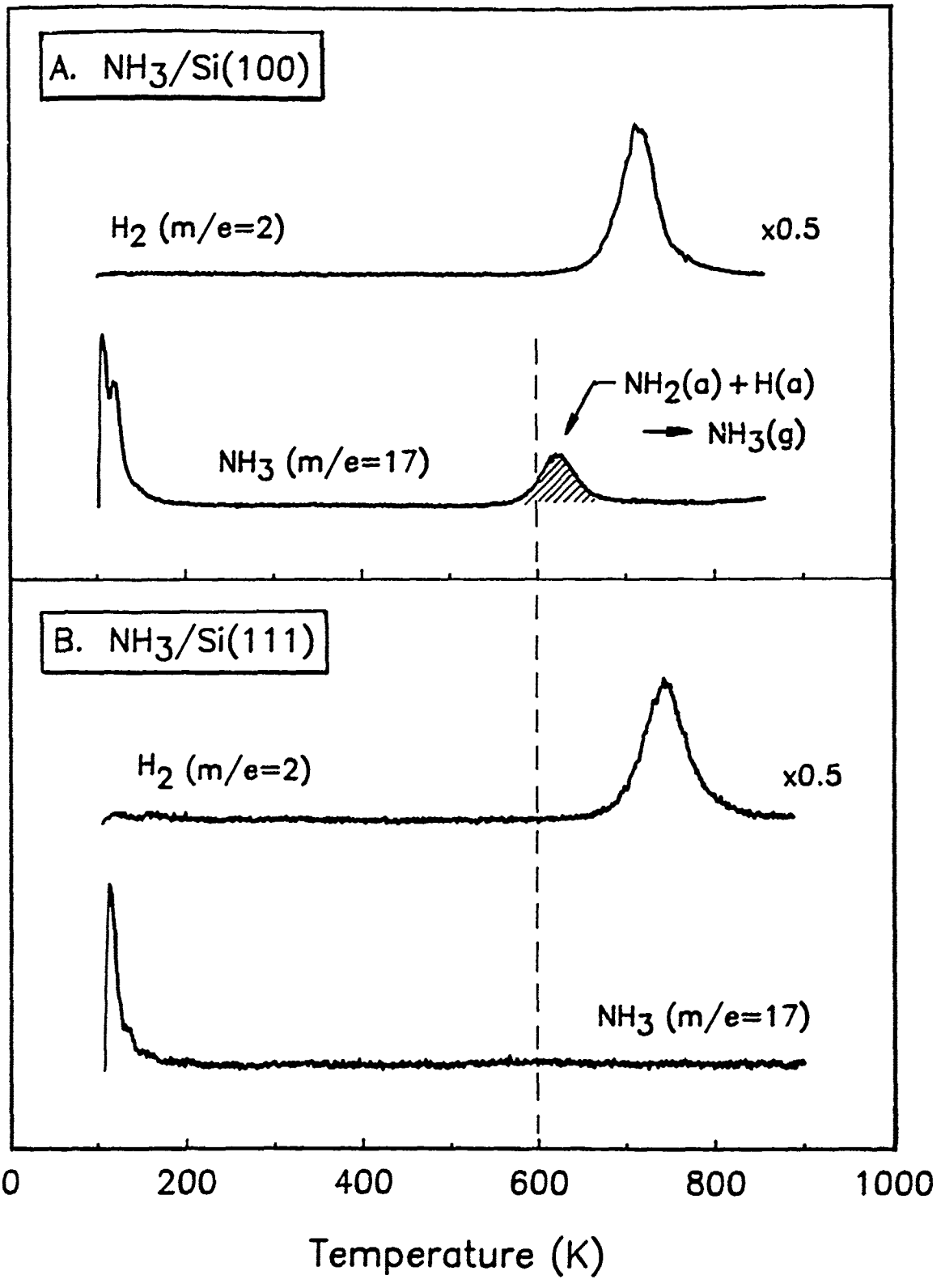

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Figure 2

Comparison of backbonding for Surface Sites on Si(111)-(7x7)

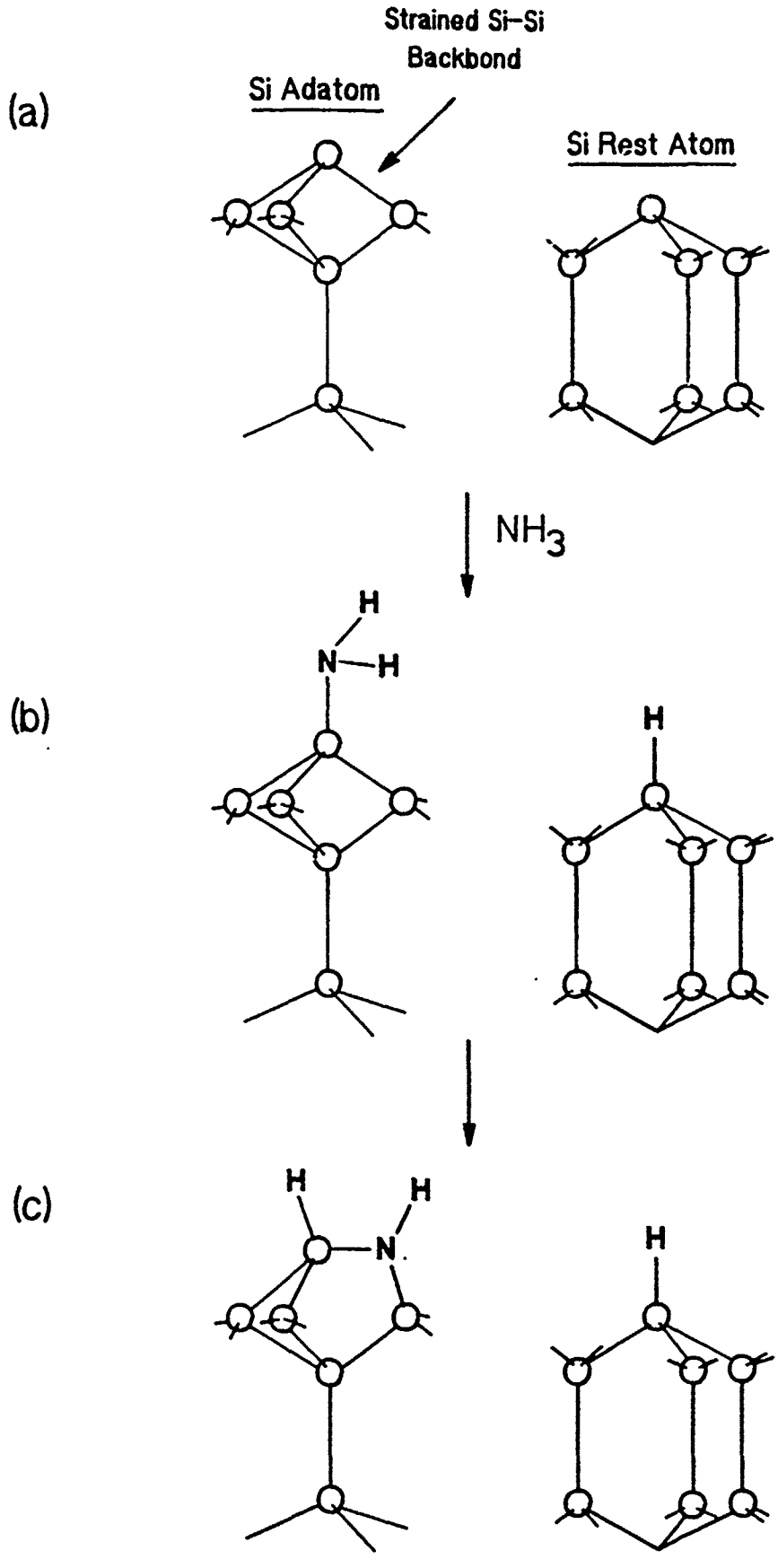

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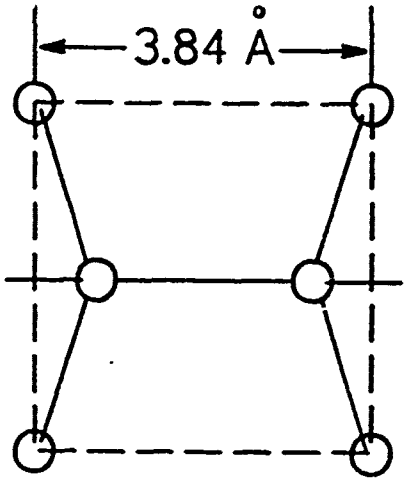
Figure 3

Surface Sites for NH_3 Dissociation on Si(100)-(2x1)

(a)



(b)

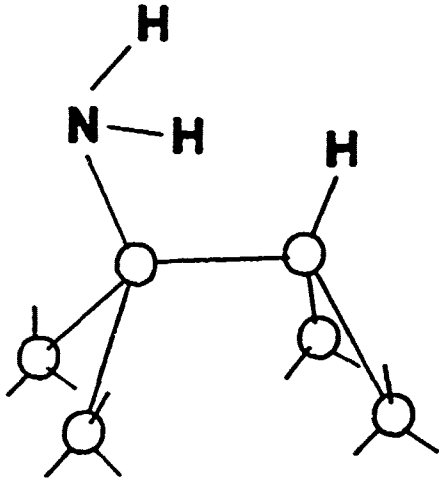


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Figure 4

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Tel: (919) 515-2377/3272
FAX: (919) 515-3419
e-mail: davis@mte.ncsu.edu

Prof. Salah Bedair 1
Department of Electrical Engineering
N.C.S.U.; Box
Raleigh, NC 27695
Tel: (919) 515-2336
e-mail: jll@@ecegrad.ncsu.edu

Max N. Yoder 1
ONR Code 1114
Arlington, VA 22217
Tel: (703) 696-4218
FAXes (703) 696-2611/3945/5383
e-mail: yoder@charm.isi.edu

Dr. A. M. Goodman 1
ONR, Code 1114
Arlington, VA 22217
Tel: (703) 696-4218
FAXes (703) 696-2611/3945/5383
e-mail: goodman@ocnr-hq.navy.mil

Dr. J. Pazik 1
ONR Code 1113
Arlington, VA 22217
Tel: (703) 696-4410
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e-mail: pazik@ocnr-hq.navy.mil
pazik%estd.decnet@ccf.nrl.navy.mil

Prof. J. T. Yates, Jr. 1
Dept. of Chemistry
Surface Science Ctr.
University of Pittsburgh
Pittsburgh, PA 15260
Tel: (412) 624-8320
FAX: (412) 624-8552
e-mail: yates@vms.cis.pitt.edu

Robert J. Markunas, R.A. Rudder 1
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Research Triangle Park, NC 27709-2194
Tel: (919) 541-6153
FAX: (919) 541-6515
e-mail: rjmk@rti.rti.org

Professor Mark P. D'Evelyn 1
William Marsh Rice University
Dept. of Chemistry
P.O. Box 1892
Houston, TX 77251
Tel: (713) 527-8101, ext. 3468
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e-mail: mpdev@@langmuir.rice.edu

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Houston, TX 770054
Tel: (713) 529-9040

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e-mail: hksionwk@ricevm1.rice.edu

Prof. A. F. Tasch 1
Dept. of Electrical Engr. & Computer Science
Engineering Science Bldg.
University of Texas at Austin
Austin, TX 78712
Tel:
FAX:
e-mail: tasch@roz.ece.utexas.edu

Prof. Charles Tu 1
Dept of Electrical & Computer Engr.
UCSD
LaJolla, CA
Tel: (619) 534-4687
FAX: (619) 534-2486
e-mail: cwt@celece.ucsd.edu

Prof. John E. Crowell 1
Department of Chemistry
University of California at San Diego
LaJolla, CA
Tel: (619) 534-5441
FAX: (619) 534-0058
email: jcrowell@ucsd.edu

Prof. P. Daniel Dapkus 1
University of Southern California
University Park
Los Angeles, CA 90089-1147
e-mail: dapkus@mizar.usc.edu
Tel: (213) 740-4414
FAX: (213) 740-8684

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